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14. ABSTRACT
Technologies that provide real-time assessment of filter impregnate residual lifetime will increase the efficiency of filter usage and ensure safe operating conditions. Detection technologies that are small, low cost, and can be located within or near filter elements are desirable. We are studying highly sensitive microcantilever sensor platforms for residual life indication. Piezoresistive static microcantilevers respond to changes in surface stress by changing resistance as the cantilever deflects from analyte interaction with the surface coating. New cantilevers

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Report Title

Functionalized MEMS Sensors for Capacity-Based Residual Life Indicators

ABSTRACT

Technologies that provide real-time assessment of filter impregnate residual lifetime will increase the efficiency of filter usage and ensure safe operating conditions. Detection technologies that are small, low cost, and can be located within or near filter elements are desirable. We are studying highly sensitive microcantilever sensor platforms for residual life indication. Piezoresistive static microcantilevers respond to changes in surface stress by changing resistance as the cantilever deflects from analyte interaction with the surface coating. New cantilevers have been evaluated for this purpose.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

Received Paper

TOTAL:

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

Received Paper

TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

Number of Presentations: 0.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

Received Paper

TOTAL:

Number of Manuscripts:

Books

Received Paper

TOTAL:

Patents Submitted

Patents Awarded

Awards

Graduate Students

NAME	PERCENT_SUPPORTED	Discipline
Ilya Ellern	0.25	
FTE Equivalent:	**0.25**	
Total Number:	**1**	

Names of Post Doctorates

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Names of Faculty Supported

NAME	PERCENT_SUPPORTED	National Academy Member
Peter J. Hesketh	0.05	
FTE Equivalent:	**0.05**	
Total Number:	**1**	

Names of Under Graduate students supported

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: 0.00

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:...... 0.00

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:...... 0.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):...... 0.00

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:...... 0.00

The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense 0.00

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Names of Personnel receiving masters degrees

NAME
Total Number:

Names of personnel receiving PHDs

NAME
Total Number:

Names of other research staff

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

Technology Transfer

FINAL TECHNICAL REPORT

August 24, 2012

Functionalized MEMS Sensors for Capacity-Based Residual Life Indicators

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Date: August 24th, 2012

1. PROJECT AND REPORT OVERVIEW

a. Technologies that provide real-time assessment of filter impregnate residual lifetime will increase the efficiency of filter usage and ensure safe operating conditions. Detection technologies that are small and can be located within or near filter elements are desirable. Low-cost devices are also necessary, since filter usage is widespread. Recognition chemistries are also required to provide both sensitivity and selectivity, since gases passing through filters may contain a wide range of components. To meet this need Sandia National Laboratories and Georgia Institute of Technology are teaming to develop microfabricated sensors for residual life indication.

b. We are studying highly sensitive microsensor platforms for residual life indication. Piezoresistive static microcantilevers (MCL) respond to changes in surface stress by changing resistance as the cantilever deflects from analyte interaction with the surface coating. Sensitivity to sub-nanogram quantities are predicted. Our research focuses on the response of active sensor coatings to battlefield contaminants, and potentially interfering compounds that are otherwise of no concern.

c. Key achievements

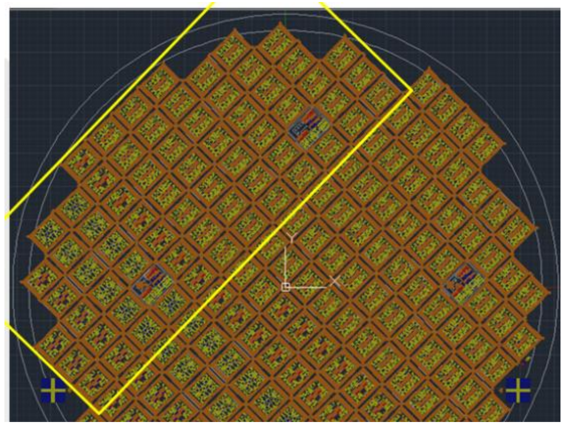
   1. New microcantilever sensor designs were fabricated in the clean room and characterized.
   2. Instrumentation was set up to evaluate the resonant frequency and piezoresistive response of cantilevers.
   3. New flow cell was set up in the lab for vapor sensing experiments.
   4. Cantilever from Georgia Tech (GT) were sputter-coated with 100nm of copper or silver and delivered to Sandia for testing.

2. PROGRESS DURING THE REPORTING PERIOD

Task 1: Fabrication of New Sensors

New fabrication process for backside etching of silicon has been demonstrated successfully
Backside etching of the silicon wafer provides effective release of cantilevers, see figure bellow. The SEM image shows a side view of an array of cantilevers on one of the silicon die after the etching has been completed. New cantilever sensors have been successfully fabricated, on 4 inch diameter SOI wafers with up to 200 die per wafer, see bellow (right)

SEM image of released cantilever beams



Die layout on wafers with 200 sensors

The electrical performance is being measured and resistance values of sensors have been, typically in the range 3600 ohms to 4100 ohms. On the 1st wafer there were 60 chips with working microcantilevers devices with good electrical performance. Die are separated from the wafer and inspected prior to wire bonding.

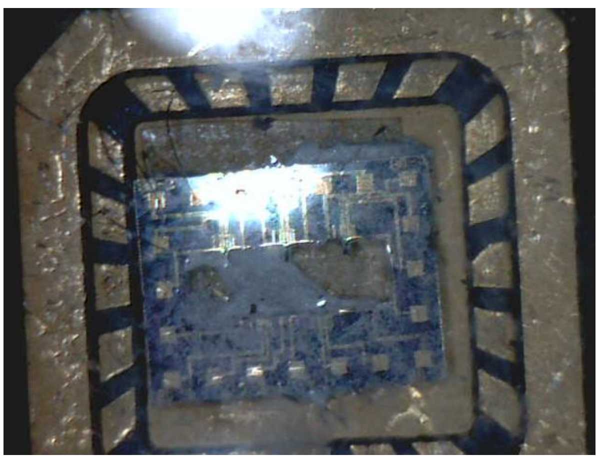


Image of one die wire bonded in a package.

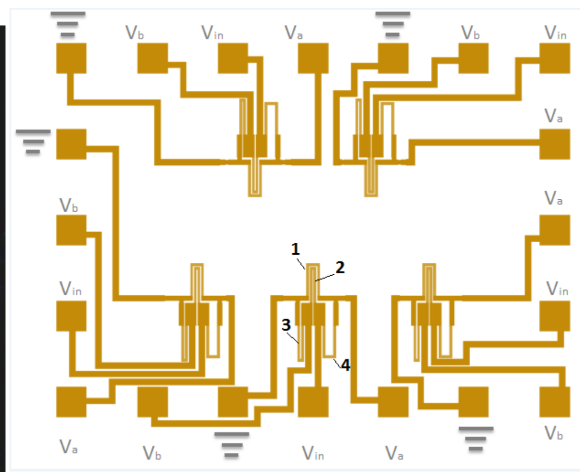
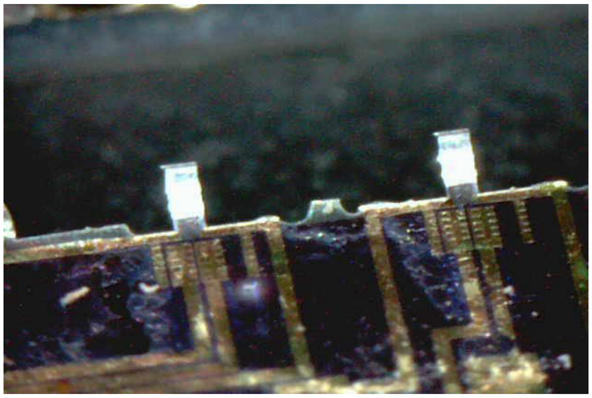
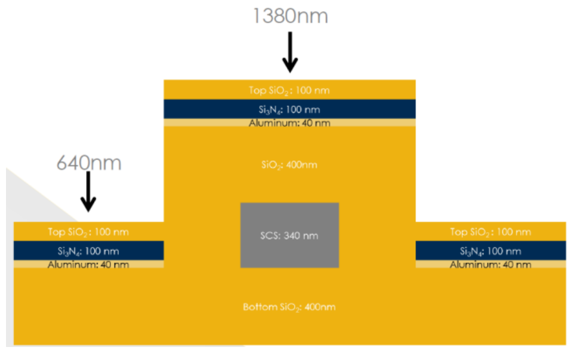


Diagram of new cantilever circuit

The photo above (left) shows one of the silicon die with multiple cantilevers mounted and wire bonded in the new test package. There are five cantilevers per die, as shown in the circuit layout above (right). Currently, the fabrication process does not yield this many fully working devices per die, however two are needed for an experiment to allow measurement of the response from a pair of coated cantilever beams. Sensors have been visually inspected and tested for electrical performance. Images bellow show two of the released cantilever sensors of the narrow beam design. Each is electrically functional, and has been coated for testing. The diagram (right) shows the cross-section of the layers making up the cantilever beam.



Released new sensors with coatings

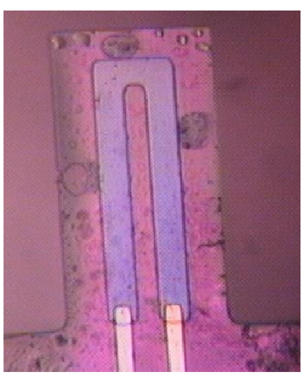


Cross-sectional diagram of layers in cantilever

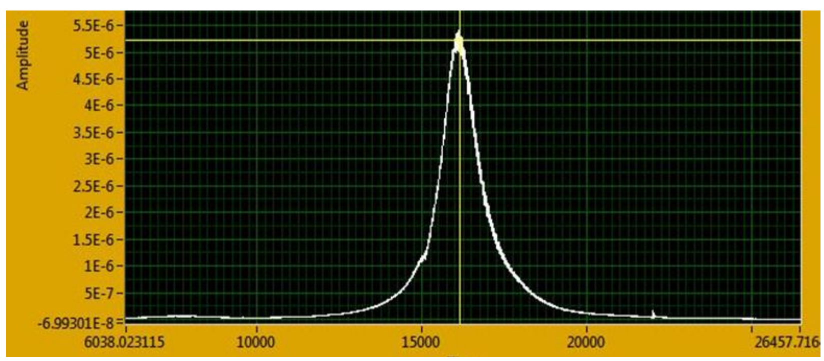
The wide beam designs, which were predicted from the modeling to have a improved sensitivity to surface stress, show a significant radius of curvature in the oxide film which will make the coating process more challenging. The next wafer of sensors is being fabricated with a stress compensation layer to achieve flat beams, as described in the later section of this report on *Stress Compensation*.

Characterization of Sensors

The resonant frequency of the cantilevers has been measured with the set up in the lab where the gas flow testing is carried out using a piezostack to vibrate the die. The Labview program make frequency scans and determines the resonant frequency of the cantilevers in the array within a few minutes, see data bellow.



Optical image of cantilever sensor before frequency test.



Frequency scan to determine resonant frequency before coating.

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Resonant frequencies of the cantilevers have been measured before and after coating with metal films. Table 1 list the resonant frequencies before and Table 2 lists cantilever frequencies after coating. The cantilevers have been coated with Copper and Silver of thickness 100nm, and have been delivered to Sandia Labs for testing.

Table 1: Resonant Frequency of Microcantilevers Before Coating

Cantilever Number	Chip N1 22	Chip N1 26	Chip N1 29	Chip N1 32
1	17459	-	22123	36571
2	-	17474	21729	17636
3	15896	11617	12676	-
4	15704	11838	12930	15486
5	14188	13746	28590	17672
6	-	-	21691	24479
7	14733	-	-	10382
8	-	15934	11135	19724
9	-	-	18878	23852
10	15821	-	19623	15497

Table 2: Resonant Frequency of Microcantilevers After Coating

Cantilever Number	Chip N1 22	Chip N1 26	Chip N1 29	Chip N1 32
1	22790	-	-	-
2	-	20446	21857	18410
3	17092	16714	15759	-
4	19544	18119	16414	20520
5	17104	16594	-	-
6	-	-	-	-
7	15638	-	-	15861
8	-	13149	-	28332
9	-	-	19561	-
10	16320	-	24286	17025

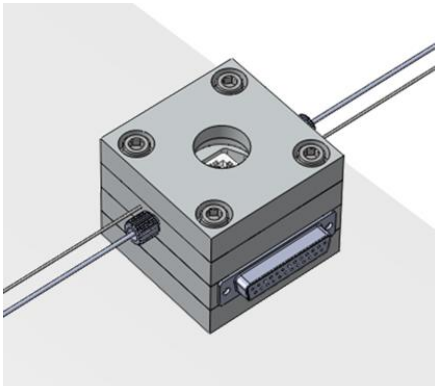
Experiments

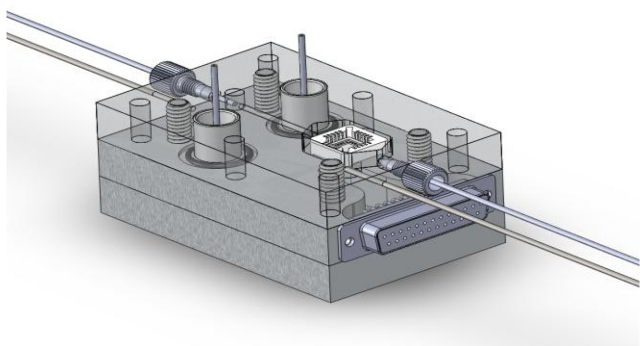
Modified experimental set-up includes temperature control of the test cell and the vapor bubbler. Multiple sensors can now be measured at one time in the flow cell and the data recorded with the Lab View program by multiplexing of the lock-in-amplifier to each of the sensor bridge circuits.

Photograph of new sensor test cell.

Photograph of new test cell with bubblers installed.

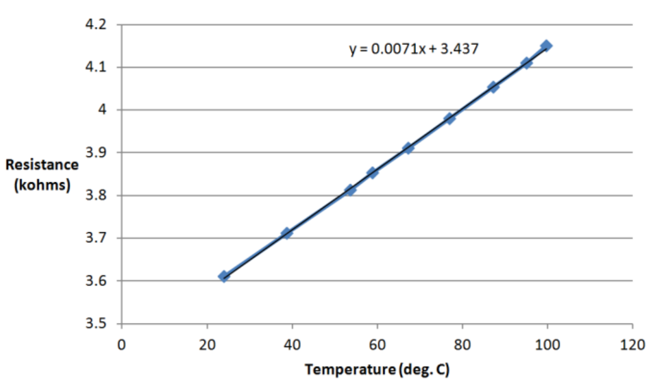
The new measurement cells have been build and evaluated. The cell (shown right) has two vapor bubblers in the cell to maintain the temperature of the bubbler at the same temperature as the test cell where the sensor is mounted. The CAD diagrams for these test cells are shown bellow.

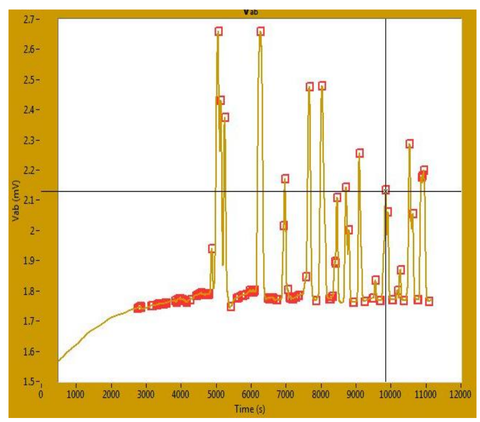




CAD file of new test cell CAD file of test cell with bubblers installed.

The new sensors have been evaluated in the cell, and the resistance as a function of temperature recorded indicating a temperature coefficient of resistance of 7 ohms per deg C, figure bellow (left). They have also been evaluated with different levels of water vapor and show a reversible response, as indicated in the data bellow (right).



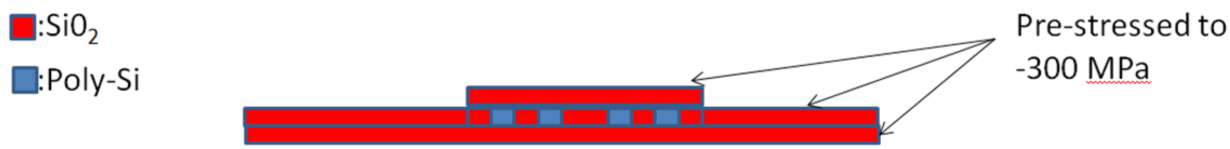


New sensors responded to flow and temperature calibration is shown (left). A MOF coated sensor responded to water vapor, see above (right).

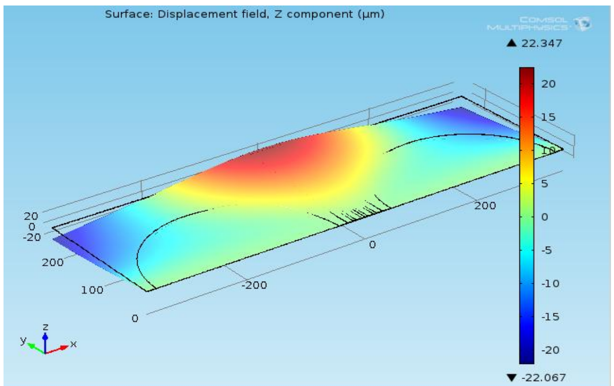
The water vapor test confirms the electrical and strain sensing of the cantilevers with a chemically adsorbing film. Cantilever sensors are now being prepared for metal coating to test the filter impregnants at Sandia National Laboratories.

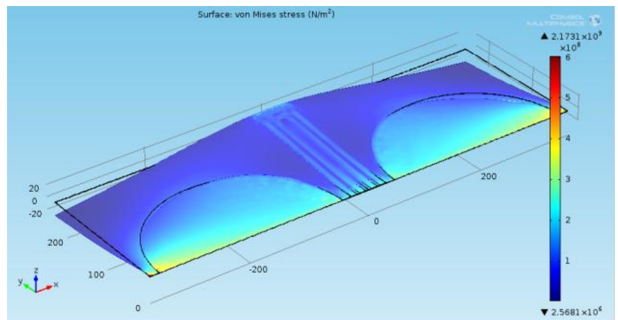
Stress Compensation

Subsequent wafers will include a stress compensating silicon dioxide film to increase the yield of working devices. Figure bellow shows the thin layers on each side of the sensing piezoresistive layer. These new devices with additional oxide coating are currently in process at the Nanotechnology Research Center clean room.



Results of COMSOL stress analysis In order the straighten the new cantilever design, a stress compensation layer is deposited on top of the beams prior to the release step. The low yield was due to stress concentration at the base of the cantilever from the compressive stress in the oxide structural layer.





The thickness of this layer has been determined with COMSOL model given the stress levels available in the PECVD systems at Georgia Tech. Calibration of the stresses has been undertaken to determine optimum thickness. For example, the Unaxis system has a compressive stress of 131MPa, and the STS has a compressive stress of 304MPa with our modified recipe. The model above shows the cantilever deflection with 300MPa stresses included in a 300nm thick film. The figures show the results for the curvature and stress compensation in wide beam designs. Hence we have the correct stress compensation film to correct for curvature and will implement this on the next wafer.

3. CONCLUSIONS

We have completed fabrication of a new design of static microcantilevers for measuring effects of exposure to battlefield contaminants. The cantilevers have been characterized and delivered to Sandia National Labs. Additional cantilevers are being prepared with stress compensation film to provide sensors to complete the testing of coated sensors with various exposure conditions, including contaminant concentration, relative humidity, and temperature. Deflection is non-reversible and progressive with continued exposure until the sensing material is consumed. It is expected that the state of an array of sensors with different coatings will be representative of the cumulative effects of these vapors on ASTM-TEDA filter media. These will serve as readily measurable imbedded residual lifetime indicators